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High Rate Cycling Behavior of the Silver Electrode

by

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ABSTRACT

A study has been made of the behavior of the silver electrod: when it is cycled at high current rates in alkaline solutions. The ratio of charge to discharge capacity has been measured at room temperature. An explanation is suggested for the inefficiency that is observed.

INTRODUCTION

Since the work of Andre (1) the use of the silver-zinc-alkali system as the basis for a secondary battery has increased rather markedly. However, the system still suffers from certain disadvantages. For example, the cycle life is usually not lengthy for appreciable depths of discharge, and the capacity often decreases with cycling. Some of these drawbacks can be traced to the individual cell components and others probably result from a combination of factors.

Another drawback of such batteries is the apparent inability to accept a high rate charge readily and efficiently. This is considered the fault of the silver electrode. This inability to accept a high rate charge presents an obstacle to the use of silver-zinc batteries in aero-space applications. During a two-hour orbit the charging time often is limited to a period of the order of 30 - 45 minutes.

Some studies have been made of this reported inability of the silver electrode to accept a high rate charge. Wales (2) has collected data under a variety of conditions such as electrolyte concentration, temperature, and current rate. He used only silver electrodes with an excess of electrolyte. A separator, not identified, was also used. With such an arrangement he found that at 25°C, using 35% and 50% KOH as electrolyte, the electrode operated with practically 100% efficiency, i.e., the discharge capacity equalled the charge capacity input. However, as the charging rate increased, the capacity accepted by the electrode before gassing took place decreased. Furthermore, the discharge current rate he used was always equal to or less than the charge current rate.

This work has recently been extended by Wales (3) to 0°C and 20% KOH as electrolyte. The charge efficiency at high current rates was below 100% in 20% KOH at 25°C, in 35% KOH at 0°C, and in 50% KOH at 0°C.

Other data have also been reported which indicate that the silver electrode will not accept a high rate charge with 100% efficiency. These studies deal with the weight gained by an electrode during charging (4) and the volume of gas evolved during charging (5). These studies indicate but do not prove or definitely prescribe the nature of this deficiency.

The work reported here was undertaken with the purpose of determining more definitely the behavior of the silver electrode during high rate charging. A high rate charge is defined here as a charge which requires an hour's duration to restore full charge after the electrode has been completely discharged.

EXPERIMENTAL

Three-plate cells were used throughout this work. These contained two negative electrodes and one silver electrode. Unless otherwise noted the negative electrodes were sintered Cd-CdO electrodes, about 4 x 5 cm. The silver electrode, 1.2 x 3.6 cm, was wrapped in a separator and inserted between the negative electrodes. Frequently a small cadmium electrode was inserted as a reference for voltage measurements. The cell cases were made of Plexiglas or Lucite. The dimensions were 9 x 5 x 2 cm. Twenty ml. of electrolyte was used but some of this was "free" electrolyte. Lucite spacers were used to pack the electrodes tightly together.

The current was supplied by a transistorized constant current source. One circuit, Figure 1, allowed a different current to be used for discharge than for charge. Voltages were fed to a pre-amp which supplied a signal to a 10 mv recording potentiometer.

RESULTS

In the first four cells that were used, the positive plates were covered with 3 layers of cellophane. Two variables were studied: (a) electrolyte concentration; and (b) particle size of the silver in the positive plate.

Cell No. 1 had 30% KOH as electrolyte and the positive was made of Handy and Harmon No. 120 silver powder.

Cell No. 2 had 40% KOH as electrolyte and the positive was made of Handy and Harmon No. 120 silver powder.

Cell No. 5 had 30% KOH as electrolyte and the positive was made of Handy and Harmon No. 130 silver powder.

Cell No. 6 had 40% KOH as electrolyte and the positive was made of Handy and Harmon No. 130 silver powder.

Before beginning the cycling regime, the cells were each given two cycles at a current of 10 to 40 milliamperes. The results of the subsequent cycles are shown on Figures 2 - 5. The current rates used are indicated on the graphs. Each cell was taken apart after these cycles and only with cell No. 2 was anything unusual detected. This cell had a broken connection to the negative terminal. The electrolytes were clear, the plates appeared to be in good condition, and the separators were heavily contaminated with silver. In all but a few instances the cells were not allowed to gas on charge. The results show little choice between 30% and 40% KOH as electrolyte at room temperature.

The plates containing the finer particles of silver, cells Nos. 5 and 6, did not give as high capacities but gave somewhat better cycle life. However, the difference is not great.

Throughout all these runs the silver electrode did not deliver as much capacity on discharge ashad been supplied in the previous charge. With few exceptions, the capacity put into the electrode during charge was greater than that delivered by the cell on discharge. Discharge was terminated at 0.75 volt vs. the Cd-CdO electrode. As expected, the capacity put into the cell during charge decreased as the charging current rate increased.

Cells Nos. 5 and 6 were cycled further to check more carefully the observation that the silver electrode accepted more capacity on charge than it delivered on discharge. Cell No. 5 was cycled by using 100 ma. on charge and 200 ma. on discharge. On charge only as much capacity was put into the cell as was delivered on the previous discharge. During none of these charges did the cell reach the gassing potential. This shows that the cell was not fully charged even at the rate used. However, the discharge capacity decreased as the cell was cycled, Figure 6.

Cell No. 6 was also subjected to further cycling using 75 ma. on charge and 150 ma. on discharge. Here again the charge input was equal only to the previous discharge capacity except for cycles 6, 9, 10, 16, and 20 (Figure 7) in which a greater amount of charge was supplied, but in no instance did the charging voltage reach that associated with oxygen evolution. The capacity tends to decrease though not as rapidly as with cell No. 5. When cell No. 6 was taken apart there were black spots on the silver electrode. Thus at the end of all these cycles this silver electrode was not completely discharged.

Two more cells, Nos. 9 and 10, were constructed similar to cells Nos. 2 and 6. In this case the positive electrode was wrapped with fibrous sausage casing, five layers on one side and three on the other side. The reference electrode was wrapped in cellophane. After two cycles at 20 - 50 ma. the cells

were cycled at 100 ma. on charge and 150 ma. on discharge. The results are shown on Figures 8 and 9.

The first 15 cycles on cell No. 10 were somewhat erratic because a meter was reading inaccurately and thus less charge was put into the cells than had been calculated. The points in Figure 8, however, are the corrected values. Again the trend is the same as that observed previously. Beginning with cycle 36 the charge put into a cell was equal to the previous discharge capacity with the exception of cycle 62. The charge on this cycle and on cycle 35 was at a low rate, 20 ma. and shows that the capacity of the cell is not permanently lost. As long as the charge capacity only equals the previous discharge capacity the capacity of the cell decreases with cycling. Cell No. 10 would accept about 375 ma-hrs. of charge before the voltage reached that of oxygen evolution.

The first ten cycles of cell No. 9 are omitted from Figure 9. These were lower current rate cycles and showed that at low rates the cell had a discharge capacity of about 400 ma-hrs. Only about one-fourth of this capacity is realized at the high current rates used in the cycles shown on Figure 9. The capacity gradually decreased over the first 25 cycles and then remained constant for the next 75 cycles. On Figure 9 the charge is shown only when it was of a different quantity from the previous discharge capacity. The results on cell No. 9 show that it is possible to have the silver electrode operate at 100% charge/discharge efficiency at the fairly high current rates used here. It is not known why this was possible with cell No. 9 but not with cell No. 10. The only difference between these cells was in the particle size of the silver used in making the silver plate. However, the data at hand are insufficient

to attribute the difference in behavior to this factor.

All these results suggest two possibilities.

1- The silver electrode accepts a charge at high charge rates but this is generally not completely available on discharge. This may be due, e.g., to pockets of silver oxide which were bypassed because more favorable current paths had been set up around them.

2- All the charge capacity does not produce silver oxide, i.e., there is some side reaction using up electricity on charge. This side reaction is not the normal production of oxygen because at no time did the silver electrode potential reach the value necessary for oxygen evolution.

There is evidence that the first possibility does explain some of the inefficiency noted here. It appears that in some instances the Ag_2O does provide a barrier to the complete utilization of the silver. For instance, a sintered silver electrode was cycled in such a way that the electrode was charged at the 15 hour rate and given only a partial discharge at about the 100 hour rate. After some 35 such cycles a complete discharge gave only about half the capacity obtained before the electrode was given these 35 cycles. Upon examination of the silver electrode it was found that the bottom third of the electrode was undischarged. X-ray examination indicated Ag_2O. An explanation for this is that during such cycling the Ag_2O builds up and some of it is by-passed on discharge because of other more favorable current paths. As this process is repeated, pockets of Ag_2O may be electrically isolated for purposes of discharge.

In cell No. 6 above, it was also noted that there were undischarged areas on the silver electrode after the cycling regime.

Attempts were made to modify the cycling behavior of the silver oxide electrodes by adding other metallic oxides. Oxides having a relatively low electrical resistance were sought with the object of providing more conducting paths through the electrode. The idea was to offset the high electrical resistance of Ag_2O and thus provide for better utilization of the available silver and avoid the isolation of Ag_2O pockets in the electrode.

The oxides studied were: MoO_2, MnO or MnO_2, ZnO, HgO, CdO, CuO. Of these only MoO_2 showed any promise. With the others, very short cycle life was obtained or the voltage characteristics were unfavorable. MoO_2 was mixed with silver powder and sintered at 1000°F for 1/2 hour. The electrode, about 1 x 2 cm, was wrapped in cellophane and cycled with two Cd-CdO electrodes in 40% KOH. Two such electrodes were prepared. Each cycle lasted 12 hours. One electrode gave 21 cycles and the other 17. The capacity decreased with cycling and the cell did not have a good shelf life. However, the electrodes held up well physically during the cycling. But the performance was not good enough to warrant further investigation at this time.

The use of palladium as an addition to the silver electrode has also been suggested, (6,7). Plates prepared with palladium additions were also cycled but showed the same behavior under high rate conditions as the untreated silver electrodes.

While the accumulation of isolated Ag_2O pockets on silver electrodes has been noted with lower current cycling and with cycling involving incomplete discharges, this has not been observed generally on any of the electrodes which were subjected to high current rate cycling. Thus, the charge/discharge inefficiency at high rate cycling cannot be explained by the formation and isolation of such Ag_2O pockets in the electrode. Furthermore, if this were the

cause it is difficult to account for the gradual and smooth decrease in capacity as cycling proceeds, Figures 2 - 7. Another explanation must be sought.

This other explanation suggests that not all the current on charge produces silver oxide. It has been suggested earlier (8) that oxygen is formed and adsorbed on the silver electrode during the charge period when AgO is being formed. This accounts for the 100% Faradaic efficiency so far as weight gain is concerned and also the fact that the charge/discharge efficiency is less than 100%. If this is the case then on discharge the adsorbed oxygen could be released from the electrode surface.

According to Figure 9 the charge/discharge efficiency at high rates decreased with cycle life up to a point and then levelled off at about 25% of original capacity. This can be explained by assuming that on charge a layer of silver oxide is first formed and that after this is formed the further formation of AgO and the production of adsorbed oxygen occur simultaneously. It may be that penetration of oxygen, or oxide ion, $O^{=}$, into the lattice then occurs much more slowly. Consequently, the charge/discharge efficiency is 100% so long as one is operating with only the initial oxide layer and becomes less than 100% as more AgO is formed.

A cell similar to those described above was assembled and sealed with an epoxy resin. An outlet from this cell was connected to a small open end manometer which contained dibutyl phthalate as the liquid. The purpose of this was to measure small pressure differences within the cell during cycling. The pressure within the cell was carefully measured at the beginning and at the end of each discharge. The cell was charged at different current rates but all

discharges were at 150 ma. This cycling was all done at room temperature which was assumed to stay constant during the relatively short time periods for each discharge.

The silver electrode contained about 1% palladium as an added impurity. The same electrode was used throughout a series of runs. The results of some of these runs are summarized on Figure 10. Different charging current rates were used and the change in pressure was measured between the beginning and the end of charge. The charge was terminated when the voltage rose to 1.55 or 1.60 volts vs. the Cd-CdO electrode. The cell was then allowed to stand on open circuit over night. After this it was discharged at 150 ma. and the change in pressure was again measured between the beginning and the end of discharge. From Figure 10, which also includes the capacity values for these runs, it is evident that under these conditions there was a small pressure increase during charge and during discharge.

There are several possible explanations for this pressure increase.

1- an increase in the temperature of the cell.

2- reaction of AgO with the electrolyte to form Ag_2O and O_2.

3- liberation of adsorbed gases from the electrodes.

The increase in pressure on charge may be due to (1) and (2) above. Furthermore, some small amounts of oxygen may be produced electrochemically during the formation of AgO on charge (5). The temperature factor is a difficult one to determine precisely since the temperature inside the cell was not measured. However, the cell was completely exposed to the air around it and there was nothing to interfere with heat exchange between the cell and the environment. Still, the effect of temperature cannot be neglected for with the cell that was used a 1° or $2^{\circ}C$ change in temperature could cause the pressure changes noted.

During discharge it is unlikely that temperature played as large a role, largely because the discharges were short. For all the cycles, except the one in which the cell was charged at 10 ma., the discharge lasted less than an hour.

Another way to account for the pressure increase on discharge is to assume a reaction between the AgO and the electrolyte. This reaction results in the formation of oxygen which would lead to an increase in pressure in the closed system. However, the cells were allowed to stand on open circuit for at least 16 hours before each discharge. During this time any initial reaction between the electrode and the electrolyte would have occurred. By the time the discharge was run the amount of oxygen produced by this reaction would have been very small (9). Then too, the discharge lasted less than one hour in all but one case. Thus the amount of such reaction taking place during discharge would be negligible.

The third possible explanation for this pressure increase during discharge is that during charge some oxygen is produced and physically adsorbed by the electrode. During the discharge this oxygen, which does not deliver Faradaic capacity, is released from the electrode and this increases the pressure within the cell case. This mechanism or process has been proposed before (5) and the results given here are consistent with that hypothesis.

The pressure changes given on Figure 10 are likely minimum values because the cadmium electrode has the ability to absorb, by reaction, the oxygen present in the cell. Such absorption would not be great here because the pressures inside the cell are but slightly different from atmospheric pressure and because there is excess electrolyte present, i.e., the cadmium electrode is operating under a "flooded" condition.

If the adsorption of oxygen on charge and its release on discharge is considered to take place, then a ready explanation is at hand for the charge/discharge inefficiency at high current charging rates. On charge the following reactions may occur.

$$2\ OH^- \rightarrow O_{ads} + H_2O + 2e$$

$$O_{ads} + Ag_2O \rightarrow ?\ AgO$$

$$Ag_2O \quad 2\ OH^- \rightarrow 2\ AgO + H_2O + 2e$$

Only the last two reactions produce capacity which is available for discharge. On discharge the adsorbed oxygen is released as the electrode surface is changed.

On Figure 10 the charge and discharge capacities are shown. For all except one run the charge/discharge efficiency is less than 100%. This exception was the last run made in this series and at this time, obviously, some previously unused capacity was available for discharge.

On the basis of the results represented on Figure 10 it appears that a possible cause of the charge/discharge inefficiency at high charging rates is the adsorption of oxygen on the electrode during charge. However, this is a likely explanation. Figure 10 offers no warrant to say that this hypothesis has been proved.

ACKNOWLEDGMENT

Special thanks are due to Mr. Alex Klooster for designing the constant current circuit used in this work.

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CAPTIONS FOR FIGURES

Figure 1. Constant Current Charging Circuit.

Figure 2. Cycling behavior of Cell No. 1.

Figure 3. Cycling behavior of Cell No. 2.

Figure 4. Cycling behavior of Cell No. 5.

Figure 5. Cycling behavior of Cell No. 6.

Figure 6. High rate cycling behavior of Cell No. 5.

Figure 7. High rate cycling behavior of Cell No. 6.

Figure 8. Cycling behavior of Cell No. 10.

Figure 9. Cycling behavior of Cell No. 9.

Figure 10. Pressure changes during high rate cycling.

LEGEND FOR FIGURE 1

R_1 - 25 ohm. The exact value and wattage depends on the current desired.

R_2 - 10K ohms potentiometer

R_3 - 50 ohms, 1 watt resistor

R_4 - and R_5 - 250 ohm potentiometer

R_o and R_d - 3 PST relays

B - 1.5 volt dry cell

T_1 - Transistor, Sylvania 2N677

T_2 - Transistor, Sylvania 2 N35

T_3 - Zener diode, Hoffman 1N1767

A - ammeter or shunt for measuring current.

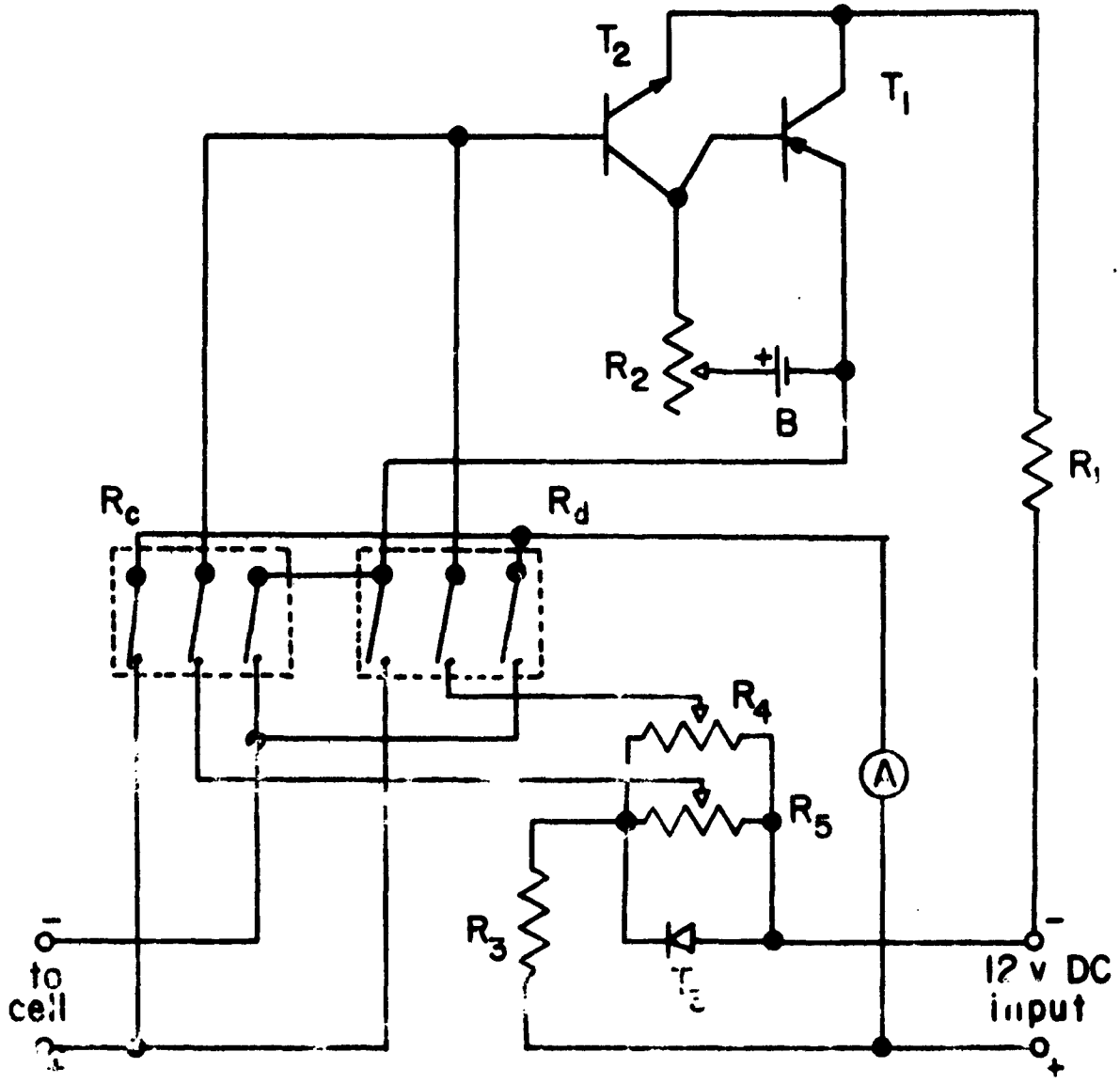


Figure 1

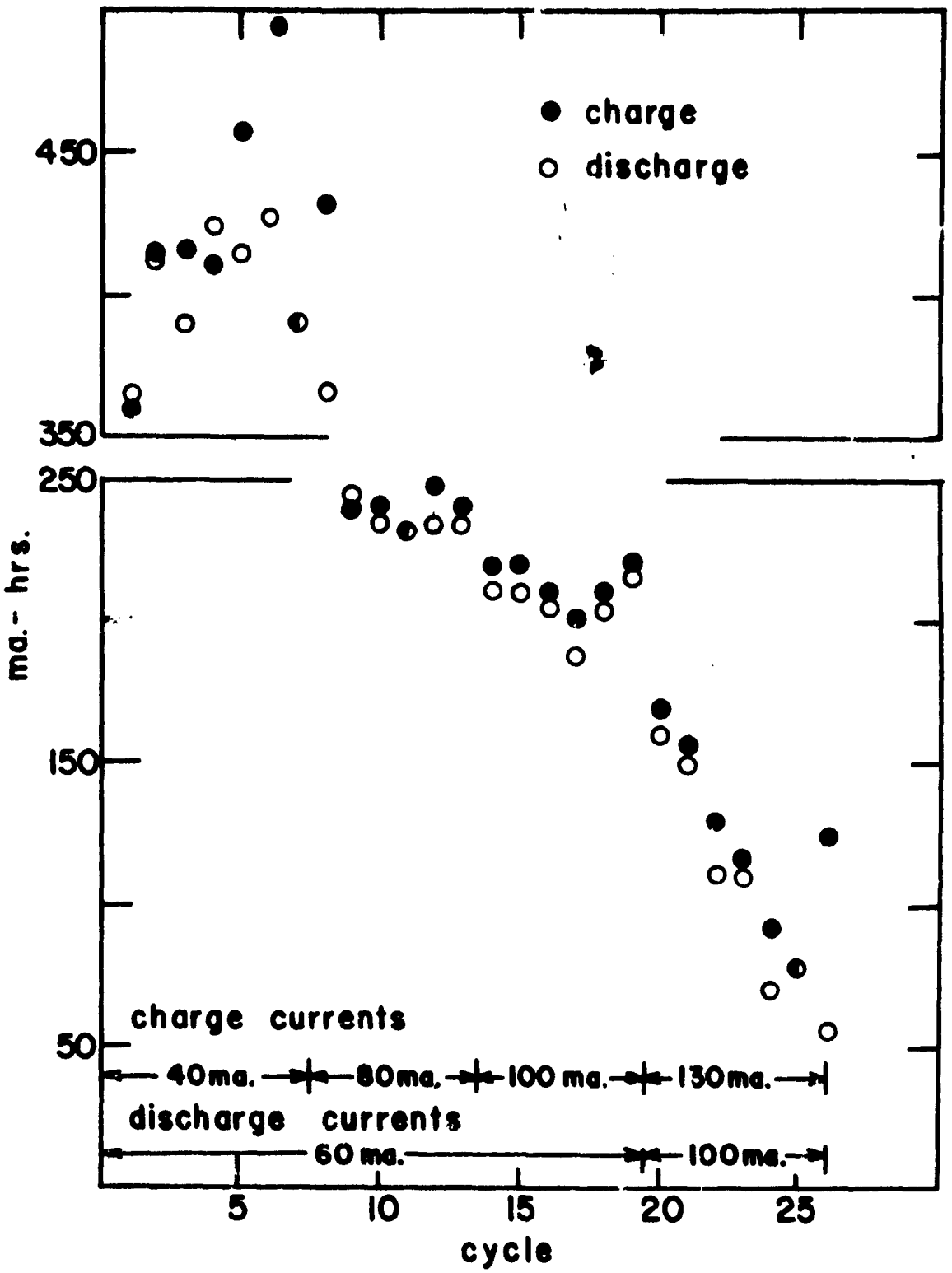

charge
discharge
450
350
250
150
50
ma.- hrs.
charge currents
40ma.
80ma.
100 ma.
130ma.
discharge currents
60 ma.
100ma.
5
10
15
20
25
cycle

Figure 2

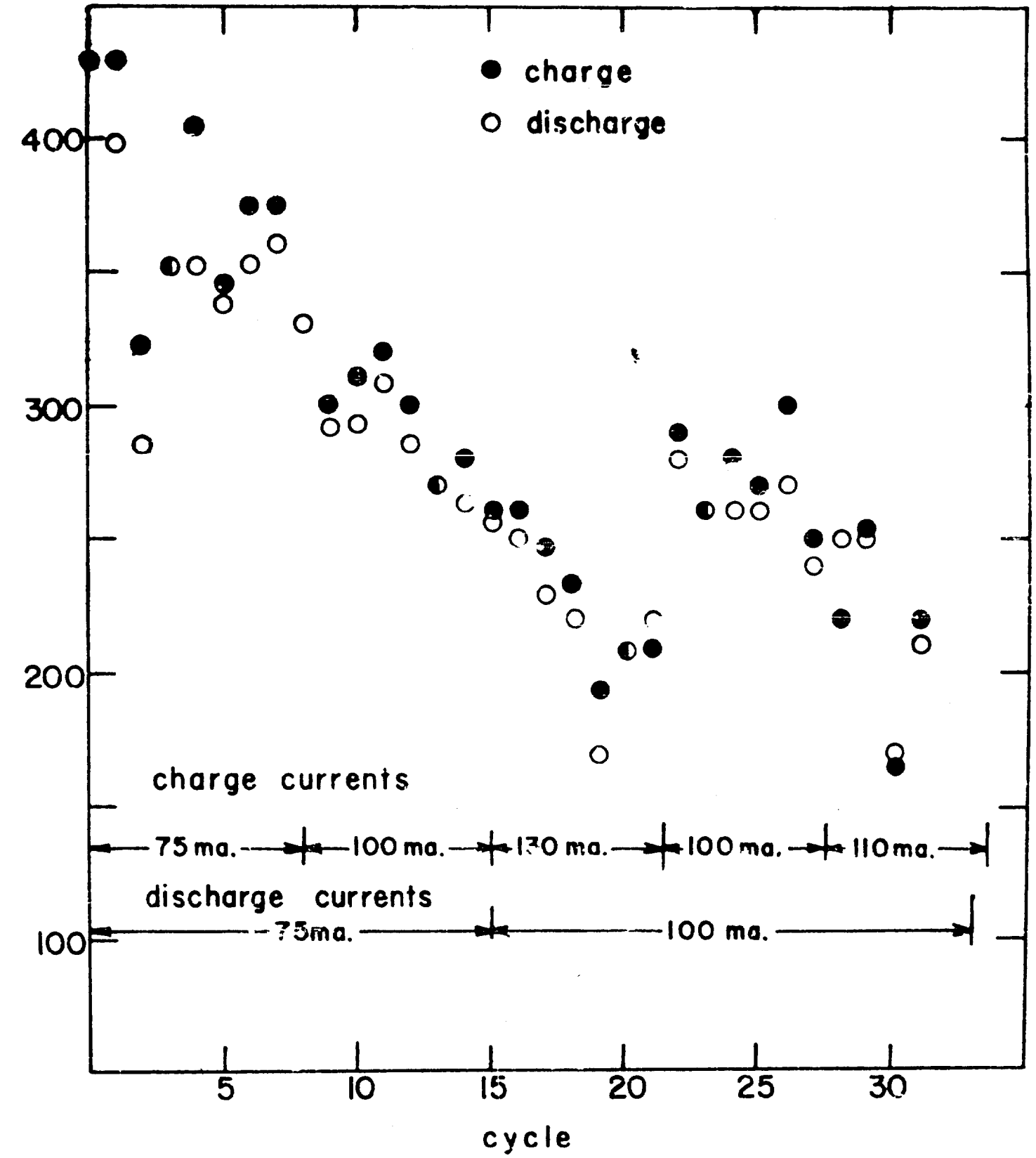

ma.-hrs.
charge
discharge
400
300
200
100
charge currents
75 ma.
100 ma.
130 ma.
100 ma.
110 ma.
discharge currents
75ma.
100 ma.
5
10
15
20
25
30
cycle

Figure 3

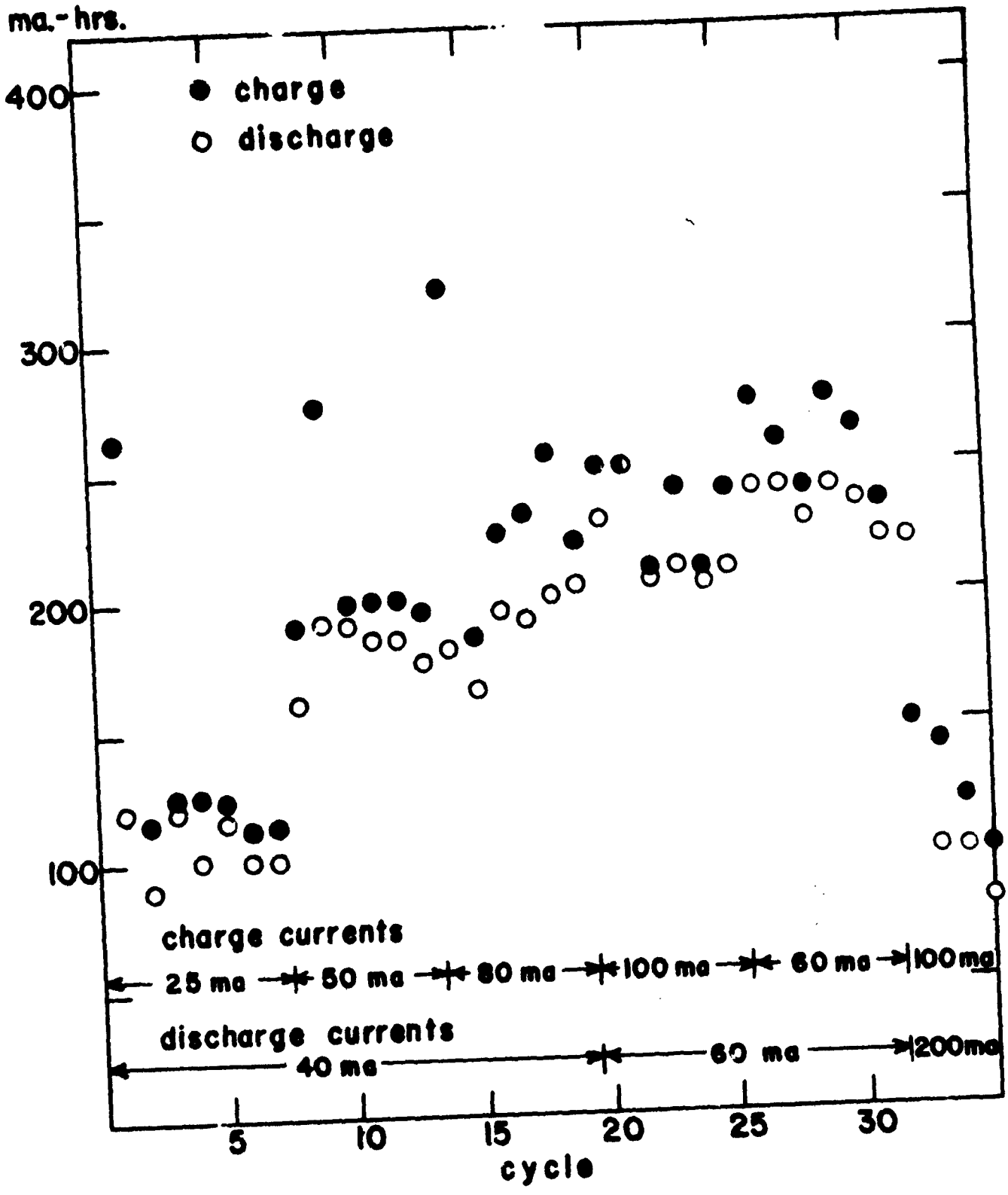
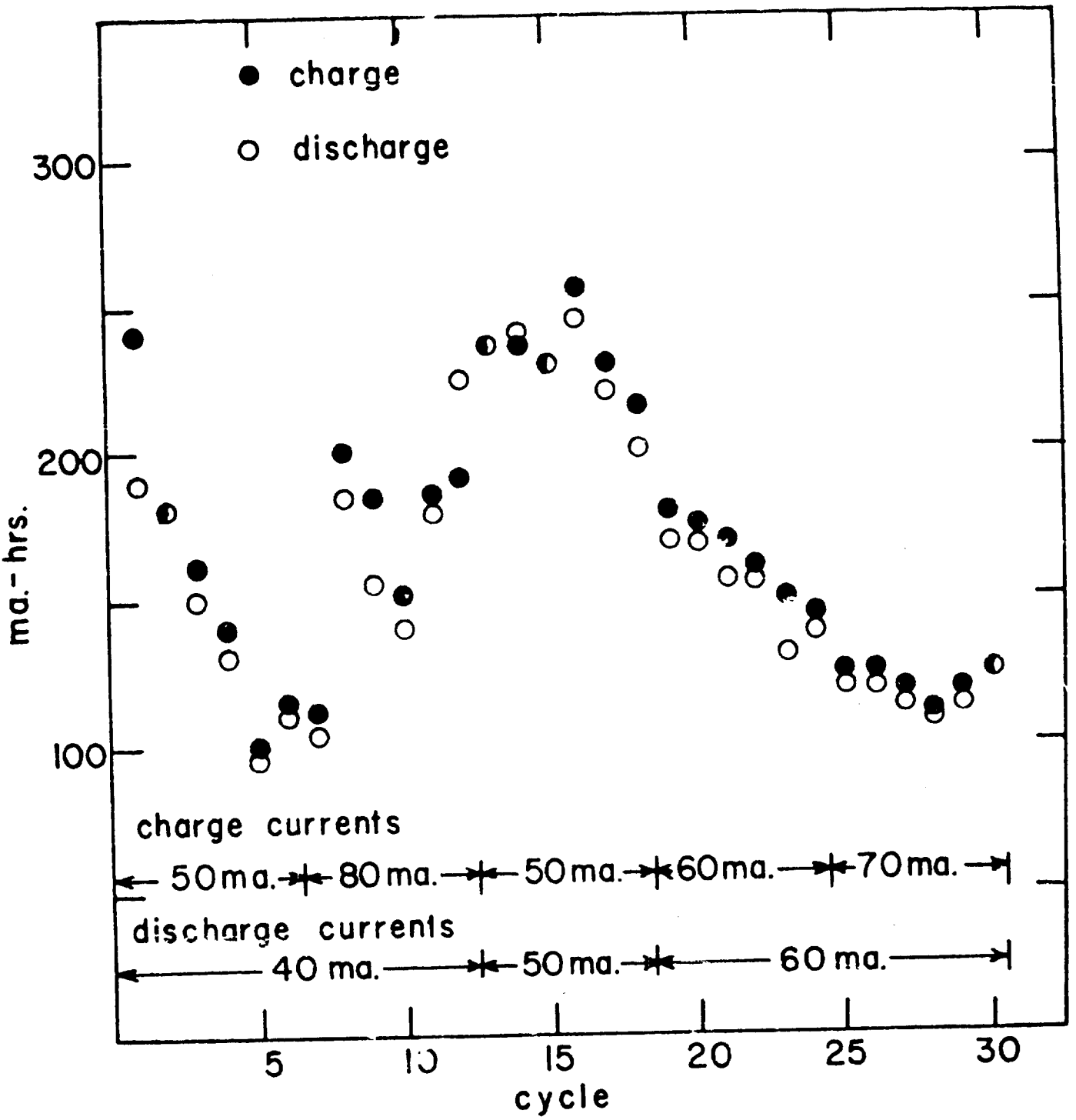

ma.-hrs.
400
300
200
100
charge
discharge
charge currents
25 ma
50 ma
80 ma
100 ma
60 ma
100ma
discharge currents
40 ma
60 ma
200ma
5
10
15
20
25
30
cycle

Figure 4


charge
discharge
300
200
100
ma.-hrs.
charge currents
50ma.
80ma.
50ma.
60ma.
70ma.
discharge currents
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50ma.
60 ma.
5
10
15
20
25
30
cycle

Figure 5

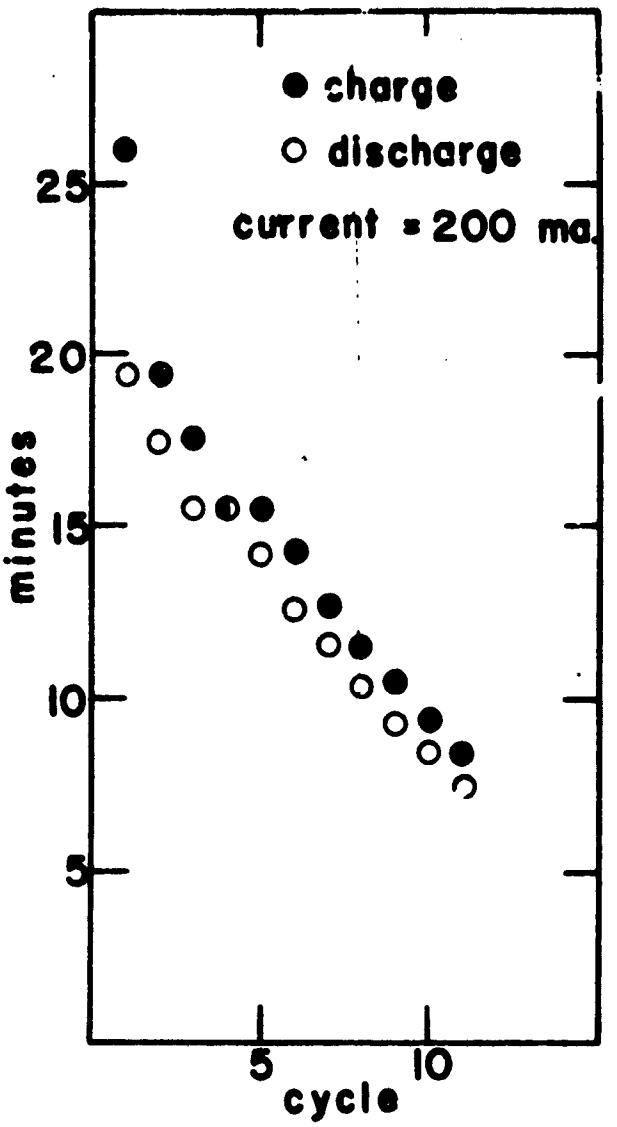

charge
discharge
current = 200 ma.
25
20
15
10
5
minutes
5
10
cycle

Figure 6

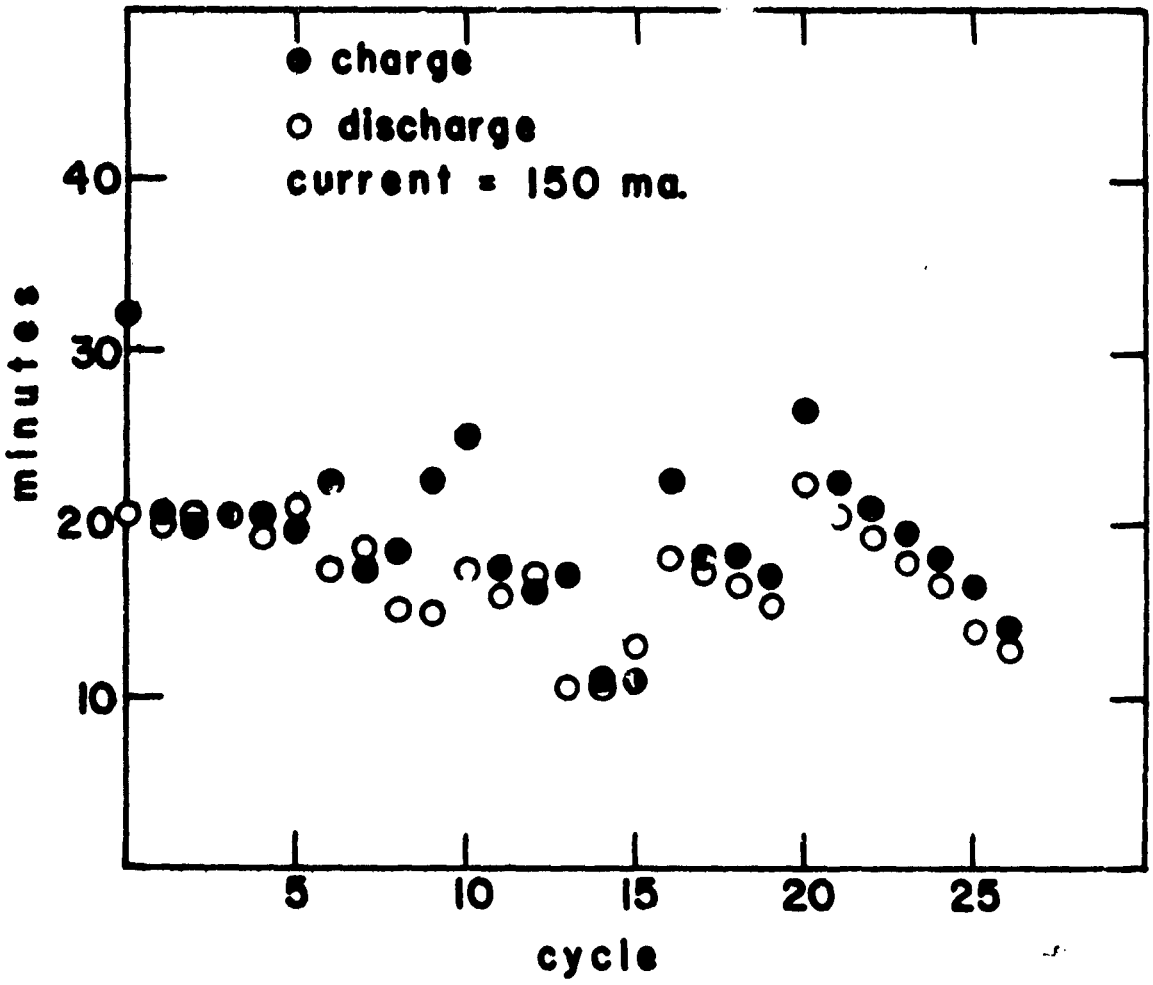

charge
discharge
current = 150 ma.
40
30
20
10
minutes
5
10
15
20
25
cycle

Figure 7

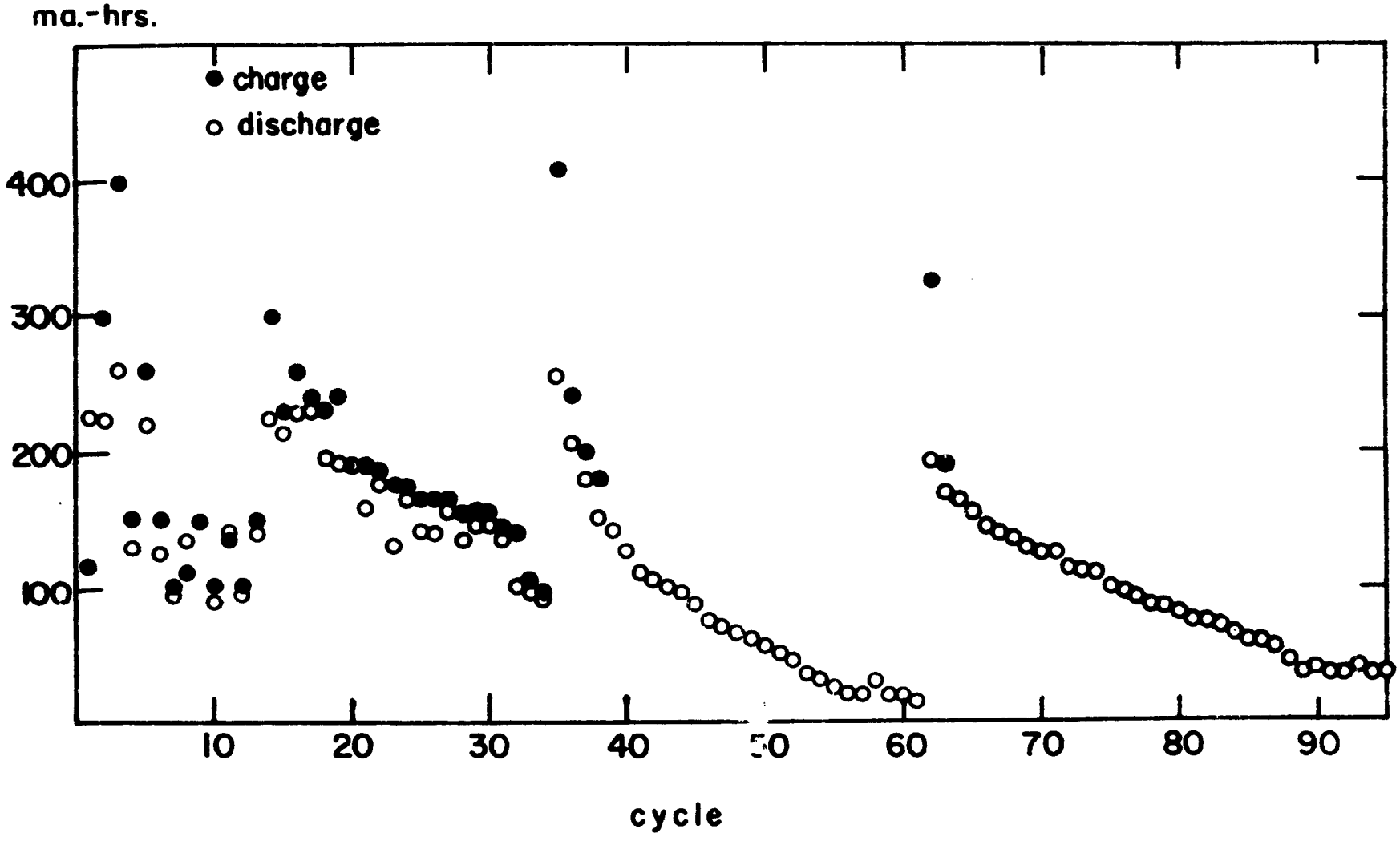
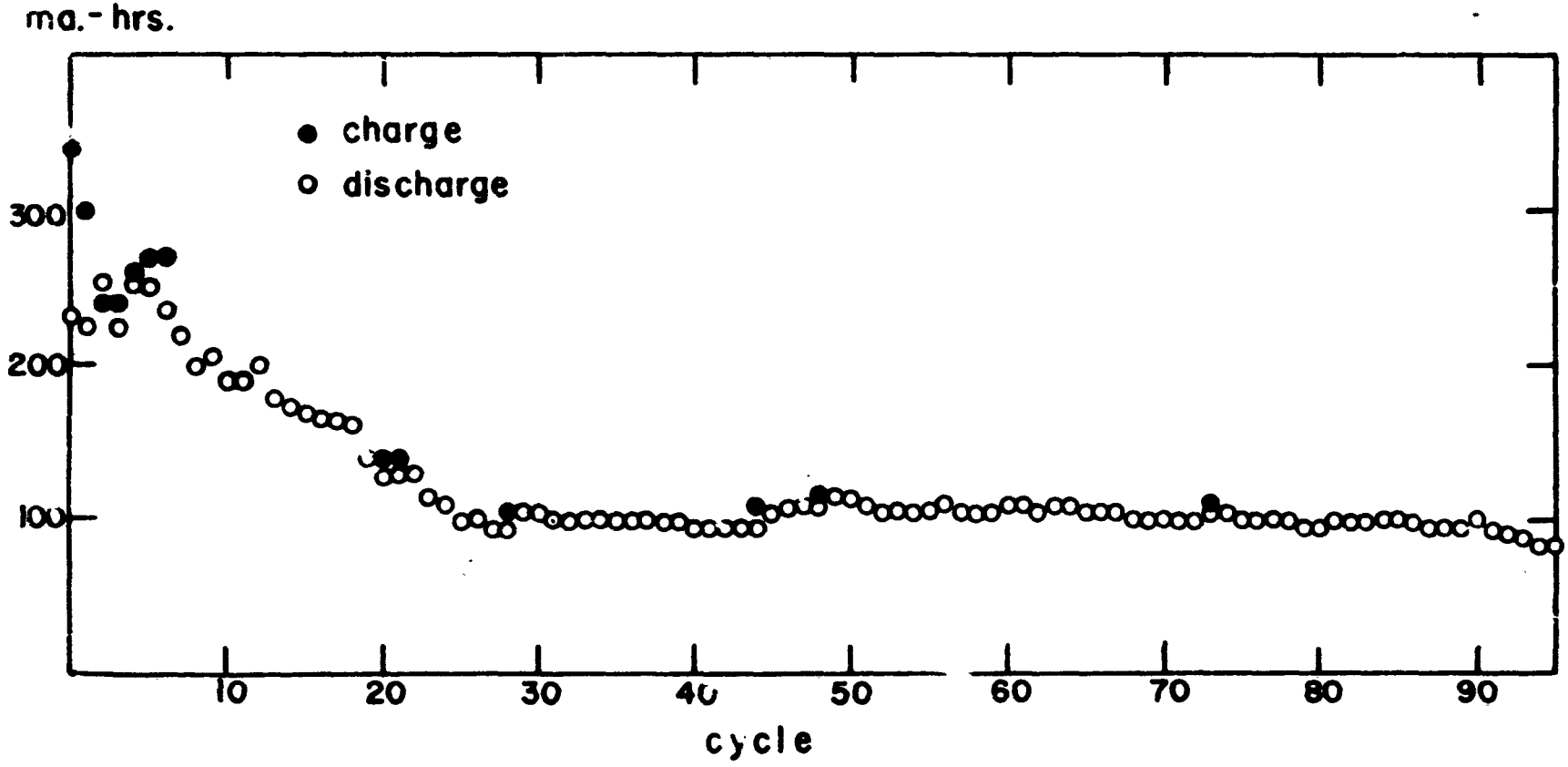

ma.-hrs.
charge
discharge
400
300
200
100
10
20
30
40
50
60
70
80
90
cycle

Figure 8


ma.-hrs.
charge
discharge
300
200
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10
20
30
40
50
60
70
80
90
cycle

Figure 9

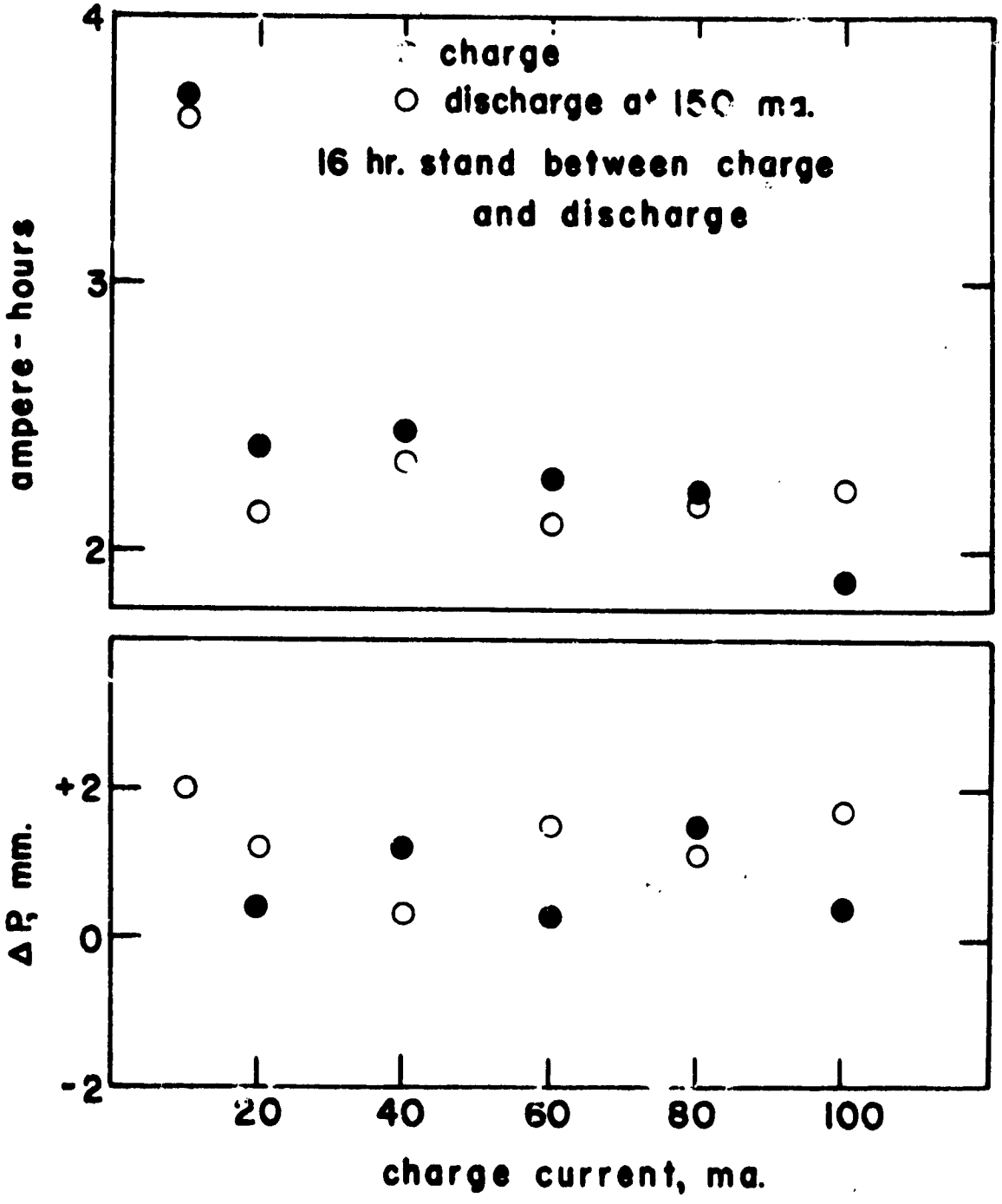
charge
discharge at 150 ma.
16 hr. stand between charge and discharge
ampere - hours
4
3
2
ΔP, mm.
+2
0
-2
20
40
60
80
100
charge current, ma.

Figure 10

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